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FORMING ALUMINUM FOR SOLAR ENERGY CONCENTRATORS

MATERIALS REPORT
FOR THE PERIOD

1 OCTOBER 1963 THROUGH 31 MARCH 1964

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FORMING ALUMINUM FOR SOLAR ENERGY CONCENTRATORS

MATERIALS REPORT

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APB 8
This report describes the tests, experiments, developments, and studies in various problem areas relating to aluminum electroforming and scale-up of the process for fulfilling the technical requirements of the NAS 1-3309 contract. The period covered is the first 6 months of the contract, 1 October 1963 to 31 March 1964. This report summarizes the technically significant efforts in solving material and processing problems.

29781 I. COMPOSITION OF THE ALUMINUM PLATING SOLUTION

Consideration was given to all alternative plating solutions reported in the literature. A hydride-type plating bath was adjudged as, presently, the most promising, as regards deposit quality and process control. Our investigation established that a 3.4M AlCl_3 - 0.4M LiAlH_4 - diethyl ether solution represented the optimum composition for the hydride-type bath.

Author

The components are defined in Table I.

II. PREPARATION OF THE PLATING SOLUTION

Various feasible procedures for preparing the plating solution for electroforming the 30" mirrors were experimentally investigated. The slow, careful addition of ether to AlCl_3 resulted in potentially hazardous localized overheating, even when external cooling was employed. On occasion, the highly exothermic heat of solution (580 calories per gram) caused an almost uncontrollable, run-away temperature rise. Slow additions of AlCl_3 to ether was the most controllable procedure for preparing the plating solution. This procedure, with adequate cooling to minimize ether volatilization losses, has been adopted for the preparation of the solution to electroform the 30" aluminum mirrors. Subsequent addition of LiAlH_4 posed no problems, although evolution of gas occurs at the start. Metal Hydrides, vendor of LiAlH_4 , indicated that this is normal and no cause for concern.

In preparing each bath, glass tanks and laboratory glassware were used to eliminate any plating bath contamination. Ether was added slowly through a teflon plug-stopcock valve. Agitation, during solution of solids, was provided by a glass-enclosed magnet in the mixing tank, with the motor and magnetic drive located outside the glove box. This worked satisfactorily and permitted use of less costly non-explosion proof equipment.

III. PLATING BATH LIFE

A. Life in Storage

One plating solution was sealed and stored for a month after preparation in glass containers. Electroforms deposited from this "aged" solution did not differ in any respect from deposits from a freshly-prepared solution. Apparently, a tightly sealed plating solution has a long life, measurable at least in months.

B. Life in Use

The solution was permitted to go to exhaustion during a planned series of deposition runs to establish bath life. After 5 weeks intermittent use, the plating bath showed signs of exhaustion: the stresses noticeably increased, and the ductility of the deposits decreased. Absorbed water was particularly detrimental to the bath. A simple control of bath condition was suggested by Dr. McGraw and tried by us. Electrode depolarization time increased with the increase of moisture content in the bath; this is easily picked up with an oscilloscope. Figures 1a, b, and c illustrate this method. The bath was subsequently restored by the addition of 0.1M LiAlH_4 .

The second, $1\frac{1}{2}$ liter plating solution was operated under conditions of continuous test and maintenance schedule. After initial deposition runs, 2 liters of a like-composition solution were added to scale up this bath, more runs were conducted, and the volume was further increased to $5\frac{1}{2}$ liters. At the end of thirteen electroforming runs, 2 more liters of solution were added, and a 7" parabolic mirror was deposited from the now $7\frac{1}{2}$ liter solution. Altogether, approximately 44% of the aluminum content of the $7\frac{1}{2}$ liter bath has been depleted. The deposits remained low-stressed and extremely ductile throughout the runs. Lithium aluminum hydride additions were made to maintain the bath composition.

IV. DEPOSITION TEMPERATURE

Our laboratory cell, contained within a glove box, has no provision for cooling, therefore, some rise in temperature resulted due to IR heating. The character of the electroform deposit was not affected within this $\pm 15^\circ\text{C}$ temperature fluctuation.

The plating solution boiling point proved much higher than anticipated. The equilibrium temperature of one deposition run was 50°C , and although the volatilization losses were high, boiling did not occur. It should be noted that pure diethyl ether boils at 35°C . A reflux condenser was incorporated into the final design, to minimize volatilization losses.

V. FILTRATION

Commonly used plating room filtering equipment cannot be used with this ethereal solution. A simple filtration unit was constructed which functions by alternately increasing and decreasing the pressure in the filter chamber. The "higher" pressure cycle was provided by dry nitrogen gas (prepurified-grade, dewpoint -75°F). A vacuum pump was used to reduce the pressure for the alternate cycle. The sub-atmospheric pressure resulted in boiling in the filter, giving excessive ether volatilization losses. This difficulty was minimized by bleeding air into the vacuum pump to reduce the vacuum to a point where boiling did not occur. The intermittent filtration during laboratory operation kept the volatilization losses within tolerable limits; however, the close watch needed with this unit would not be practical for scaled-up operation.

The filtration operation can be illustrated by referring to Figure 2. Plating solution enters the glass filter chamber during its reduced pressure cycle until a sufficient solution height is reached. The column is then pressurized to force the solution through the glass cloth filter bed. Glass check valves maintain flow in the proper direction.

The filtration unit for the scaled-up equipment was designed so that the pressure varies between 6-10 psig and atmospheric pressure (high and reduced pressure cycles, respectively). These conditions have been checked out in the laboratory with complete success, operationally and with respect to ether losses.

Couch and Brenner (Ref 2) stated that the plating solution does not require frequent filtration, since the insoluble impurities and black anode slime settle to the bottom of the laboratory cell and usually do not cause rough deposits. This contradicts the data from our runs, in which filtration was omitted. Rough nodular deposits were obtained without filtration, when the aluminum electroform thickness exceeded 0.008 to 0.010-inch without current reversal, or exceeded 0.012 to 0.014-inch with current reversal. With repeated filtration during deposition, the number and size of nodules sharply decreased when the proper current reversal cycle was employed, even at deposit thickness in excess of 0.030-inch. This is extremely important in our program, since the nodules on the rear surface show through to the front. Continuous filtration will be used in electroforming the 30" aluminum mirrors.

VI. AGITATION OF THE PLATING SOLUTION

A glass-covered magnetic stirrer was used to provide mechanical agitation of the plating solution during several deposition runs. This agitation was observed to stir up anode sediment, making filtration more difficult, and resulting in a roughened deposit. Agitation was reluctantly omitted from subsequent deposition runs.

In electroforming the 30" mirror, where some agitation is considered mandatory, agitation will be provided by having the filtered solution, returning from the filter, impinge directly on the male nickel master. In addition, a slow rotational motion of the male nickel master will result in gentle secondary agitation and serve to make the deposited thickness more uniform.

VII. ELECTRODES

A. Anode

2S* aluminum anodes have been used during deposition to maintain bath purity at acceptable levels. The anode geometry has been flat plate and, to date, no attempt has been made to use conforming anodes, even with the hemispherical or parabolic electroforms. The throwing power of the electrolyte (ability to plate geometrically remote regions) proved to be surprisingly high. However, a conforming anode will be used for the large mirrors to insure greater uniformity of deposited thickness. The anode will be shrouded in glass cloth to "contain" the black anode slime and reduce the amount of "pore-clogging" sediment to the filter. Glass cloth was determined superior to various alternative materials cited in Ref 3 (i.e., alundum, cellulose pulp, polyethylene, felt paper, various rubbers) in compatibility with the plating solution, sufficient porosity for deposition, strength, non-deterioration, pliability, availability in various sizes and cost.

* Sometimes designated 1100 aluminum. Purity: 99.0% min. Aluminum, to 1.0% Si and Fe, 0.20% Cu, 0.05% Mn, 1.10% Zn.

B. Cathode

Aluminum electroforms have been deposited successfully on nickel, copper and brass substrates in the laboratory. The primary emphasis, however, has been with electroformed nickel substrates to simulate to 30" electroformed male nickel master. Accordingly, various sizes of flat electroformed nickel tools were utilized for deposition. Subsequently, parabolic (5" and 7") and 2½" hemispherical electroforms were deposited from corresponding electroformed or plated nickel masters to simulate the ultimate geometry and substrate material. No difficulties were experienced in any of these runs.

VIII. PRETREATMENT OF CATHODE SUBSTRATE

Successful electroforming requires that the deposit exhibit sufficient adhesion to prevent premature parting during deposition, and still permit easy separation at the conclusion of the run, so that the electroform need not be pried off and thus distorted.

For the initial ("shakedown") run, the cathode substrate received no pretreatment. The subsequent deposit, although extremely thin, peeled away from the substrate. Many complex, costly substrate treatment procedures are cited in the literature (Ref 1, 3 and 4). A simple procedure utilizing an oleic acid solution was developed in the laboratory and is used in conjunction with a thin chemically-reduced silver film applied to the substrate. The drawback of this method is the adherence of the silver film to the electroformed aluminum; however, it permits distortion-free separation of the soft aluminum deposit, which is quite significant when considering the problems generally encountered in pulling off the much more rugged nickel electroforms from their masters. After about a month's storage in a desk drawer, the silvered surfaces show signs of tarnish. Methods to remove the adhering silver film without damaging the geometry and reflectivity of the aluminum substrate or omit the silver film from the procedure are under investigation.

IX. CURRENT DENSITY

The character of deposit for a range of current density (0 to 140 asf) was investigated utilizing General Electric's proprietary Schmidt Stress Cell. The deposit from just one run permits evaluation of the entire current density range cited above. Blistering, and some peeling, was noted at less than 15 asf, whereas, at high current densities, the deposit turns rough. A relatively wide current density range (15-35 asf) was found to be suitable for our requirements. Aluminum electroforms deposited within this range (generally about 20 asf) were highly satisfactory.

X. CURRENT REVERSAL

The smooth deposits which can be achieved with current reversal have been reported by Couch and Brenner (Ref 2). Our laboratory investigation came to the same conclusion. Short d plating cycles were not effective. A 294-second plating, 6-second deplating time cycle produced excellent results for our process conditions. The 6-second (2% time) deplating cycle proved optimum in preventing nodule growths, while not greatly reducing the overall plating efficiency.

The effect of not using current reversal in electroforming "thick" deposits is the increased tendency to form nodule growths which show through the front surface. This was shown pictorially in Ref. 5.

XI. POWER FAILURE

Anticipating possible power failure during the deposition of the 30" aluminum mirrors, the consequences and possible corrective procedures of planned current interruptions of varied duration, were evaluated in the laboratory cell. Power shut-downs or removal of the electroform from the plating solution caused delaminations in the deposit. Corrective procedures proved ineffectual, so that in order to prevent possible mishaps during the electroforming of the 30" mirrors, an auxiliary power supply was incorporated into the equipment design, which would maintain the master cathodic during failure of the plating current. This technique was demonstrated in a lab run, when the current density was reduced to a trickle for 8 hours. The deposition run was later resumed, and the resultant electroform was devoid of laminations.

XII. THICKNESS

Conner and Brenner (Ref 3) recommended the addition of methyl borate to the plating solution to attain deposition thickness. In the laboratory cell, aluminum electroforms have been deposited to thicknesses of 0.029-inch for a flat plate, and in excess of 0.030-inch for a 7" parabolic mirror without bath additives or any difficulty. This was encouraging, since additives, like most bath contamination, increase the stress levels in the aluminum deposits.

XIII. PHYSICAL PROPERTY TEST SPECIMENS

A flat aluminum plate. 0.029 inch thick, was electroformed, simulating the planned deposition conditions for the 30-inch aluminum mirrors. The aluminum plate was subsequently milled into strips (for thermal expansion tests) and machined into tensile specimens. All machining proceeded without delaminations of the material or any other difficulties occurring. The pulled tensile specimens broke "clean" with no delaminations of the deposit revealed. This confirms the integrity of the aluminum electroforms.

A. Mechanical Properties

Four standard tensile specimens, with a reduced cross-section of 0.406-inch x 0.026-inch, were machined from 0.028-inch electroformed aluminum plate. One specimen was used to determine the proper stress and strain magnification scales. Data from the remaining three are reported below.

Stress-strain curves (Ref 5) were obtained from tests conducted on a Floor Model TT Instron Testing Machine, using an F-cell for load pick-up. The cell was adjusted to 200 pounds full chart reading. Strain was measured with a Tinius-Olsen S-3 extensometer with a one-inch gauge length, adjusted to produce a 125x magnification. The total elongation was measured over one-inch gauge marks with a vernier calipers. The strain rate of each test was approximately 0.05 inch per inch per minute at the start of the test.

Data reduction of the stress-strain curves yielded the following average values: Modulus of Elasticity - 7.98×10^6 psi; Tensile Yield Strength @ 2% Offset - 7810 psi; Ultimate Tensile Strength - 11050 psi; and Elongation - 26%. All mechanical property data are tabulated and compared with handbook values of 2S aluminum in Table 2.

B. Thermal Expansion

Four $3\frac{1}{4}$ -inch long, $\frac{1}{2}$ -inch wide specimens were machined from 0.28-inch electroformed aluminum plate. One specimen was used for set-up, therefore, data for the remaining three specimens are reported.

The more common dilatometer method of measuring thermal expansion was impractical for the soft-thin, 0.026-inch electroformed aluminum specimens. Therefore, an alternate method was devised. A model KE-2 Keuffel and Esser Theodolite was mounted at a known distance from the specimen to measure the angle subtended by a 3-inch gauge length of the electroformed aluminum specimen. The change in gauge length was calculated from the measured change in the subtended angle which resulted from the temperature variation. The accuracy of the theodolite is within one-half second of arc.

The test specimens were taped to an aluminum block to prevent buckling. However, the adhesive side of the tape was covered where it could exert any restraint of the specimen. The specimens were placed in a Missimer High-Low Temperature Chamber and sighted during test through a 10-inch x 10-inch viewing port.

The selected temperature range of -70°F to $+200^{\circ}\text{F}$ was divided into three increments: -70°F to 0°F ; 0°F to $+100^{\circ}\text{F}$; and $+100^{\circ}\text{F}$ to $+200^{\circ}\text{F}$.

Measurements were made 30 minutes after the specimen temperature had stabilized for each temperature cited above. A shielded thermocouple mounted on the gauge length surface of the specimen monitored the temperature during these tests.

An average value of 13.6 in/in/ $^{\circ}\text{F}$ over the -70°F to $+200^{\circ}\text{F}$ temperature range was measured for the linear coefficient of thermal expansion of electroformed aluminum. These data are tabulated in Table 2.

C. Density

Two specimens were deemed sufficient because of the routine and simple character of the density determination.

The density of the electroformed aluminum specimens was determined by the standard ASTM method. All weighings were done with an analytical balance accurate to within 0.1 milligram. The specimen volume was determined by measuring the water displacement weight of the sample.

Values of 97.4 and 98.9 percent of theoretical density were measured for the two electroformed aluminum specimens. These data appear in Table 2.

D. Optical Properties

Flat aluminum plates, approximately 2 3/8" x 2 3/8" x 0.016" thick, were electroformed over an electroformed nickel tool. Deposition of the aluminum was by our optimized operating conditions which are part of our large equipment design. One electroformed aluminum specimen was mechanically polished. This electroform, a second specimen in the as-removed condition, and the nickel master from which these replications were made, were sent to Libbey-Owens-Ford for vacuum coating with aluminum and silicon monoxide, successively. These specimens were required to re-test previous samples, which were vacuum coated with an excessive (pink) thickness of SiO in-house. The specular reflectivity data of the latter specimens have been reported previously and will not appear in this report.

The specular reflectivity of the three repeat optical test specimens is measured at 100°F over the spectrum of 0.3 to 7.0 microns. A Beckman Model DK1L Reflectometer is used to determine the specular reflectivity in the 0.3 to 2.7 micron spectral range. For the 2.0 to 7.0 micron spectral range, the optical test specimen is measured in a Perkin-Elmer Model 205 Reflectometer. Reflectivity of an aluminum mirror standard is simultaneously determined to enable calculation of absolute values for each specimen.

The specular reflectivity of the electroformed aluminum specimens and nickel mirror appears in Table 3. These data indicate that mechanical polishing of the optical surface prior to vacuum coating markedly reduced the specular reflectivity in the 0.3 to 1.5 micron wave length range. The specular reflectivity of the non-polished electroformed aluminum specimen was comparable to the nickel master over the entire spectral range (0.3 to 7.0 microns).

Liberty Mirror indicated that the silver film is not a desirable substrate for their coating process.

XIV DISPOSAL OF PLATING SOLUTION RESIDUES

To date, no attempts have been made to dispose of any sizable quantities of plating solution. However, disposal of small quantities of plating solution (about 2 liquid ounces each bottle), used for compatibility studies, solution adhering to the electrodes, etc., has been performed without difficulty. The aluminum electroforms are rinsed thoroughly in fast-running water after removal from the bath to react all adhering plating solution. A 2-gallon jar of water is kept in the hood for quick immersion of the anode assembly. The small quantity of plating solution, added slowly in small increments to the 2 gallons of water, did not heat up the water excessively. Concurrently, portions of the plating solution were left exposed in the hood to reduce the volume through evaporation and slowly react with the moisture and oxygen of the air. For the small quantity of solution, this latter technique is not necessary, although this would be employed for disposal of the bulk of the plating solution if necessary.

XV. MATERIALS COMPATIBILITY

In an effort to reduce fabrication costs in the 30" electroforming equipment, the compatibility of selected standard materials was investigated. High cost of glass-lined equipment recommended this study, the results of which appear in Table 4.

The materials of construction specifications for the scaled-up equipment are based on this table.

XVI. DEFINITION OF THE 30" MIRROR ELECTROFORMING EQUIPMENT

A flow diagram, showing all equipment and process line requirements for electroforming the 30" mirror, was prepared. This was based on lab results, and considering all available information (experimentally re-checked, where doubtful) in the published literature, and after discussions with vendors and people experienced in this or like processes. A schematic of the electroforming equipment appears in Figure 3 and should be referred to for the discussion of the planned operation.

A. Preparation of Plating Solution

Anhydrous diethyl ether will be pumped by pressurized dry nitrogen into the argon-purged solution storage tank. Additions of solids are made from the glove box while a gas-driven stirrer agitates the solution. Cooling coils control the solution temperature during the exothermic reaction of solution. The solution will be recirculated into the storage tank through the filter before being pumped to the plating tank.

A condenser in the gas vent line was inserted to minimize ether volatilization losses. A pressure relief valve maintains the proper tank pressure. A rupture disc (silver foil) was included in a vent by-pass line as an emergency precaution.

B. Start-Up of the Plating Cell

The conforming aluminum anode, properly shrouded with glass cloth to retain the anode slime and minimize contamination of the plating solution, will be mounted in the empty plating tank. The solution return line from the filter passes through a centrally located hole in the anode. The plating tank will be purged with argon prior to start-up. The plating solution can now be pumped through the filtration system from the solution storage tank into the plating tank. The filtration system to the plating tank will be thus checked out before deposition. A plating tank cover will seal the tank, and a positive pressure will be maintained therein.

The glove box will then be unclamped and removed so that the male nickel master (cathode) can be attached to the hoist mechanism within the glove box. The entire assembly (male master within the glove box) is then placed in position over the plating tank, clamped down and purged thoroughly with argon. Sparkproof electrical connection is made to the cathode and anode, and the male nickel master is quickly lowered into the plating solution while the plating current is on. Current density is then adjusted to 20 asf, and the plating solution will start circulation through the filter unit.

The operation of the filtration apparatus has been described in previous progress reports. In this design, a pressure relief valve will be utilized to reduce the internal filter pressure. A vacuum pump was used during laboratory operation, but was difficult to control because of the boiling of ether in the vacuum cycle. The modification has proven better workable by test.

C. Operating Conditions During Deposition

The following plating parameters will be used during the aluminum electroforming operation:

Anode-cathode	Parallel, at a distance of $2\frac{1}{2}$ ", the anode to remain stationary.
Solution	Aluminum chloride (3.4M), lithium aluminum hydride (0.4M) in diethyl ether.
Temperature	20-30°C (heating by the plating current)
Filtration	50 gal/hr, continuous.
Agitation	Provided by filtration and cathode rotation.
Cell Voltage	10-16 Volts
Cell Current	110 to 160 amps.

Direction, control tests and additions will be provided by RSD.

Agitation will be provided by the filtering system, which will impinge the solution on the cathode through a hole at the center of the anode.

In addition, a slow rotational motion will be imparted to the male master to provide additional agitation and obtain a more uniform thickness in the 30" aluminum electroform.

Periodic current reversal will be utilized. The planned cycle will be: Plating - 494 sec; Deplating - 6 sec. RSD will supply the current reverser unit. Removal of a filter unit from the process line for cleaning includes a blow-off line, which will be used to purge the unit. Additions during operation will be made through the solution preparation tank.

D. Termination of the Deposition Run

At the conclusion of the deposition period, the plating current and filter operation will be halted. The male master with the adhering 30-inch mirror will be hoisted up into the glove box and permitted to drain into the plating tank. The plating tank cover is now replaced, and the solution is stored therein. (In an emergency situation, rapid removal of the plating solution from the plating tank can be made to either the storage tank or outside the plant (i.e., dumped into a deep ditch). Return to the storage tank could also be made through the filtration unit.)

The glove box assembly is now unclamped and hoisted away from the closed plating tank. The male master with aluminum electroform will be removed and set down near a water drain. The assembly will be washed with a heavy stream of water to react the adhering plating solution. After separation of the first 30" aluminum mirror, the male master surface will be prepared for electroforming the second mirror. Run start-up procedures will be as indicated previously.

XVII. STRUCTURAL ANALYSIS

Structural analysis was performed, as a step in complying with the contract requirement for investigation into methods of incorporating the deposits into a practical solar concentrator design.

The results of an analysis for the problem of stress and deformation of a paraboloidal shell of revolution subject to a uniform gravity field parallel to its axis appear in Ref 1. This simulates a thin shell, supported by a torus, under launch loads. Because a pure membrane solution cannot be obtained for this shell, that solution was achieved in two steps, i.e., membrane plus bending solutions.

The calculations for a particular configuration are quite lengthy. The results from a computer solution for the 30" mirror, based on the measured physical properties of electrofed aluminum, appear in Ref 5. The initial estimate of 30 mils mirror thickness, calculated from existing flat plate analysis, proved sufficiently conservative for the specified 10g axial and 2g transverse load conditions.

REFERENCES

- Reference 1 Schmidt, FJ, "Forming Aluminum for Solar Energy Concentrators"; Quarterly Progress Report No. 1 (1 Oct-31 Dec 1963).
- Reference 2 Couch, DE and Brenner, A, "A Hydride Bath for the Electrodeposition of Aluminum"; J. Electrochem Soc, 99, p 234-244 (1952).
- Reference 3 Connor, JH and Brenner, A, "Electrodeposition of Metals from Organic Solutions"; J. Electrochem Soc, 103, p 657-662 (1956).
- Reference 4 Beach, JG and Faust, CL; "Electroclad Aluminum on Uranium"; J. Electrochem Soc, 106, p 654-659 (1959).
- Reference 5 Schmidt, FJ and Hess, IJ, "Forming Aluminum for Solar Energy Concentrators"; Quarterly Progress Report No. 2 (1 Jan-31 Mar 1964).

TABLE 1

Purity of Constituents of Aluminum Plating Solution

A. Aluminum Chloride

Vendor: Fisher Scientific Co., King of Prussia, Penna.

 AlCl_3 , anhydrous, sublimed, reagent-grade AlCl_3 99% min.

Typical Impurities:	Heavy metals (as Pb)	0.002%
	Iron	0.01%
	Sulfate	0.005%
	Substance not ppt'd by NH_4OH	0.10%

B. Lithium Aluminum Hydride

Vendor (sole source): Metal Hydrides, Inc., Beverly, Mass.

 LiAlH_4 95% min.

Typical Impurities:	LiCl	2.0-2.5%
	LiAlO_2	1.0-20%
	$(\text{C}_2\text{H}_5)_2\text{O}$	0.1-0.2%
	AlH_3	0.1-0.2%

C. Diethyl Ether

Vendor: Fisher Scientific Co., King of Prussia, Penna.

Ethyl ether, anhydrous, reagent-grade

Maximum Limits of Impurities:	Residue after evaporation	0.0005%
	Water	0.01%
	Alcohol (ethylalc)	0.01%
	Acid (as acetic)	0.0005%
	Aldehyde	about 0.0005%
	Peroxide	0.00005%
	Preservative	0.00000%

TABLE 2

MECHANICAL PROPERTIES OF ELECTROFORMED ALUMINUM

TENSILE PROPERTIES

Specimen Number	Modules of Elasticity (psi)	Ultimate Tensile Str. (psi)	Tensile Yield Str. @ 2% Offset (psi)	Elongation (%)
1	7.83×10^6	10,850	7,880	24
2	8.17×10^6	11,290	(a)	28
3	7.95×10^6	11,000	7,740	(b)
Average	7.98×10^6	11,050	7,810	26
2S Aluminum	10.0×10^6	13,000	5,000	35

LINEAR COEFFICIENT OF THERMAL EXPANSION

Specimen Number	1st Run	2nd Run	Averages
1	13.2×10^{-6} in/in/°F	14.6×10^{-6} in/in/°F	13.9×10^{-6} in/in/°F
2	13.8	12.9	13.4
3	14.1	12.7	13.4
Average	-	-	13.6
2S Aluminum	-	-	$(12.2 - 13.1)10^{-6}(c)$

DENSITY

Specimen Number	
1	2.64 g/cc (97.4% of theoretical density)
2	2.68 g/cc (98.9% of theoretical density)
Average	2.66 g/cc (98.2% of theoretical density)
2S Aluminum	2.71 g/cc

(a) Erractic extensometer readings in yield area

(b) Failed through radius area before full elongation was reached

(c) Alcoa Aluminum Handbook cites 12.2×10^{-6} in/in/°F from -58° to +68°F and 13.1×10^{-6} in/in/°F from 68° to 212°F.

TABLE 3

SPECULAR REFLECTIVITY OF ELECTROFORMED ALUMINUM

All specimens were vacuum coated with aluminum and with silicon monoxide at Libbey-Owens-Ford

<u>Wave Length, microns</u>	<u>Electroformed Aluminum</u>		<u>Nickel Master</u>
	<u>As Deposited</u>	<u>Polished Surface</u>	
0.3*	71.1 (83.1)***	31.4	85.8
0.4	71.2 (87.3)***	45.7	87.7
0.5	82.6 (88.1)***	56.9	89.0
0.6	84.8 (88.2)***	64.2	88.3
0.9	87.2	75.1	88.0
1.2	93.7	86.5	94.1
1.5	95.6	90.9	96.8
1.8	95.8	93.1	96.7
2.1	96.5	94.3	96.7
2.4	96.3	95.6	96.8
2.7	96.0	96.4	97.4
2**	95.3	94.3	96.2
3	95.3	96.0	97.0
4	95.9	95.9	96.6
5	96.2	96.7	97.4
6	95.8	97.2	97.7
7	96.0	96.4	97.8

* Reflectivity determined with Beckman-Model DK1L Reflectometer for spectral range of 0.3 to 2.7 microns.

** Reflectivity determined with Perkin-Elmer Model 205 Reflectometer for spectral range of 2-7 microns.

*** Cutting of the optical specimen resulted in distortion from true flatness and erroneously high diffuse reflectivities at the low wave lengths. Specular reflectivity was determined by subtracting the diffuse reflectivity from the total reflectivity, so that this discrepancy was noted. The value in parenthesis represents an adjusted true value.

TABLE

RESULTS OF MATERIALS COMPATIBILITY TESTING

Weighed specimens were placed in closed bottles containing hydride bath for a duration of 18 days. Specimens extend above liquid level. Weight change and appearance is recorded.

	<u>Item</u>	<u>Weight Change</u>	<u>Remarks</u>
METALS	Inconel	None	Excellent appearance
	301 stainless (17:7)	None	Excellent appearance
	6061T6 Aluminum	-0.15%	Etch pits on face, edge; Vapor attack-some in liquid
	2024T3 Aluminum	-0.13%	Etch pits on edge; Vapor attack-some in liquid
	Silver	None	Excellent appearance
	Copper	None	Excellent appearance
	Nickel (electroformed)	None	Excellent appearance
	25 Aluminum foil	+0.9%	Small weight of spec.
PLASTICS	Teflon tubing	+0.27%*	Excellent appearance
	Polyethylene tubing	+2.6%*	Excellent appearance
	PVC**	-0.025%*	Excellent appearance
	Lined fiber glass**	-0.12%*	Excellent appearance
	Tygon tubing	--	Section in liquid embrittled, discolored; dissolved some; section in vapor looks good.
RUBBERS	Neoprene chunk 1/2"x1/2"x3"	--	Black color gone from section in liquid to 1/8" penetration, vapor attacked less.
	Rubber tubing	--	Swelling, discoloration in liquid-minimum or no vapor attack
	Rubber sheeting	--	Roughened appearance from liq.-min. or no vapor attack
	Viton	--	Swelling in liquid; min. or no vapor attack
MISC	Plater's tape	--	Liquid severely attacks, some attack in vapor-some "wick action"
	Plain Masking tape	--	As above, more "wick action" but less vapor attack
	Synthesine coating unfired	--	Blistered, soft, scrapes off easily
	Synthesine coating fired (400°F-20 min.)	0.11%*	Excellent appearance
	Painted Ag lacquer on Ni ***	--	Coating intact but blistered, lifts off readily

*** Shorter duration soaks planned

** Specimens supplied by a plating vendor

* Specimen returned to bath for longer exposure

TABLE 4 (cont)

ADDITIONAL RESULTS OF MATERIALS COMPATIBILITY TESTING

Weighed specimens were immersed in sealed bottles containing the hydride plating solution for a duration of 18 days. After this initial exposure, the specimens were removed, weighed and then returned to the sealed bath for an additional 18 days, after which they were again weighed. All specimens extended above the liquid level during the exposure.

<u>Material</u>	<u>1st 18-day period</u> <u>Weight Change</u>	<u>2nd 18-day period</u> <u>Weight Change</u>	<u>Remarks</u>
PVC*	-0.025%	-0.07%	Excellent appearance
Lined fiberglass*	+0.12%	-0.18%	Excellent appearance
Teflon tubing	+0.27%	+0.30%	Excellent appearance
Polyethylene tubing	+2.6%	+2.55%	Slight discoloration
Synthesine coating (fired, 400°F - 20 min)	-0.11%	-0.21%	Excellent appearance
Mild steel**	-0.02%**	----	Slight rust in localized areas**

* Specimens supplied by a plating vendor.

** Only one 18-day exposure to the plating bath.

Weighed specimens were immersed in sealed bottles containing the hydride plating solution for a duration of 34 days. The specimens were removed, weighed and the results tabulated below.

<u>Material</u>	<u>Weight Change</u>	<u>Remarks</u>
Araldite 6020 Teta loaded with MgO, Silica Microballoons	+0.094%	Excellent appearance
Araldite 6020 Teta heavily loaded with Silica Microballoons	+0.39%	Excellent appearance
Araldite 6020 Teta heavily loaded with Al ₂ O ₃	-0.0081%	Excellent appearance
Araldite 6020 Teta	+0.076%	Excellent appearance
RSD Series 124 Epoxy	+0.83%	Excellent appearance
Product Research Corp. Proprietary formulation	-	Portion submerged in liquid dissolved

FIGURE 1 - DEPOLARIZATION TIME

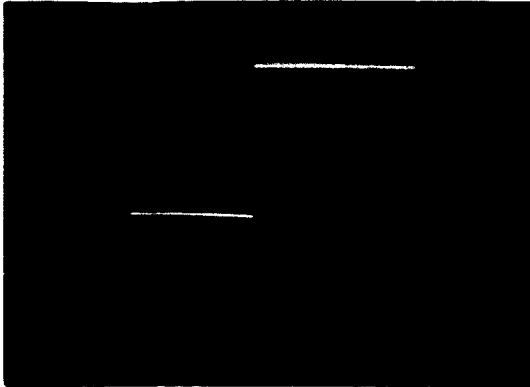


Fig. 1a - Time photograph of oscilloscope voltage measurement at the interruption of Run 12. Vertical scale: 5 volts/major division. Horizontal scale: $\frac{1}{2}$ second/major division. Power interruption dropped the voltage from 25 to 0 volts instantaneously.



Fig. 1b - Time photograph of oscilloscope voltage measurement at the interruption of Run 13. Vertical scale: 5 volts/major division. Horizontal scale: $\frac{1}{2}$ second/major division. Power interruption reduced voltage from 15 volt. Note: voltage trace exhibits a discernable difference from zero volts.



Fig. 1c - Time photograph of oscilloscope voltage measurement performed at the interruption of Run 14. Vertical scale: 2 volts/major division. Horizontal scale: 2 sec/major division. A black flakey deposit now occurs. Note: voltage did not return to zero within the 8 seconds time recorded in the photograph.

FIGURE 2: ELECTRICAL AND FILTRATION SYSTEMS TO PLATING BATH

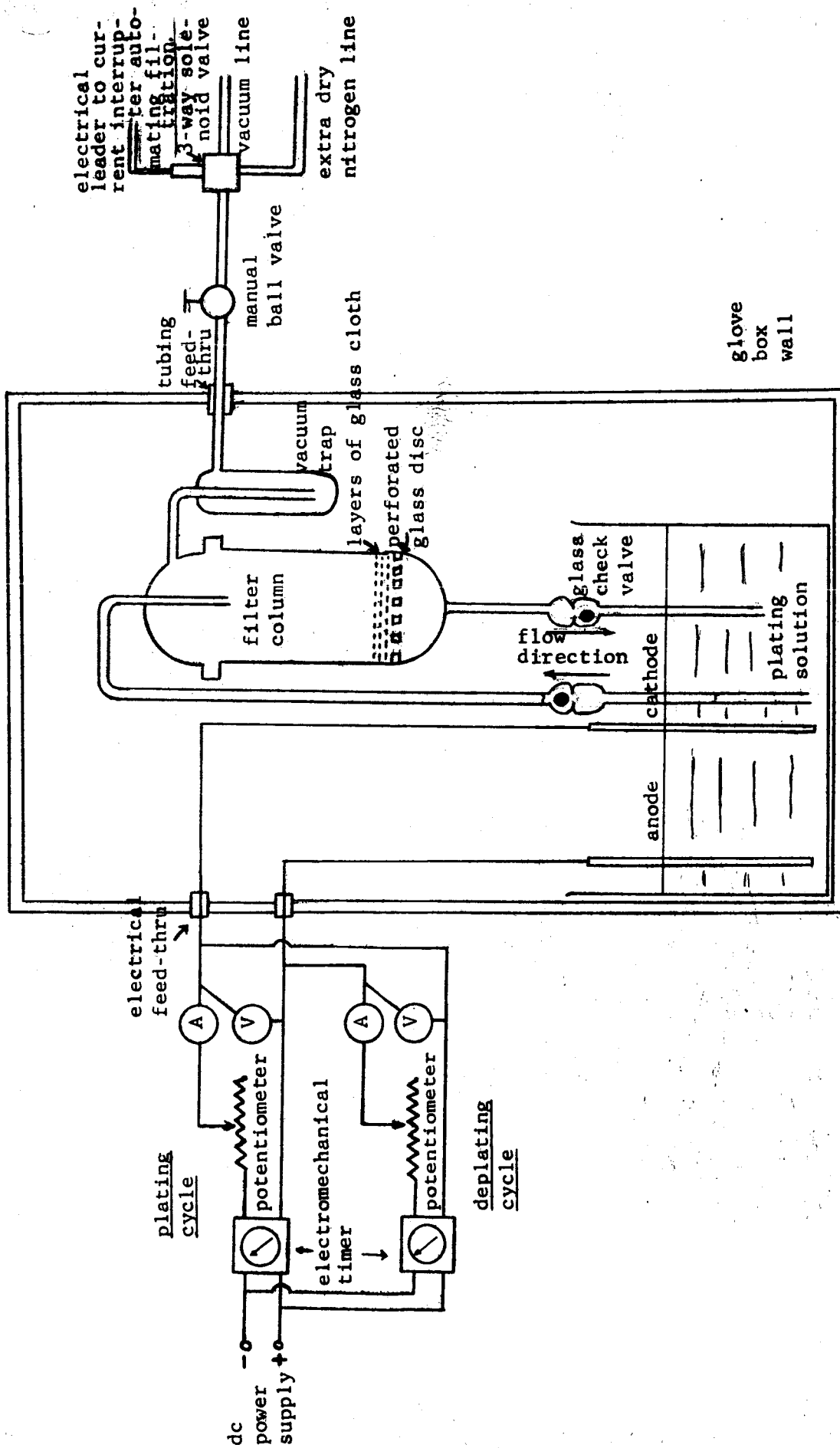


FIGURE 3 ALUMINUM ELECTROFORMING EQUIPMENT

